



AFRL-RX-WP-TP-2008-4043

DETECTION OF INCIPIENT THERMAL DAMAGE IN POLYMER MATRIX COMPOSITES (PREPRINT)

Eric Lindgren, John Welter, Shamachary Sathish, and Erik Ripberger

Nondestructive Evaluation Branch

Metals, Ceramics & Nondestructive Evaluation Division

DECEMBER 2006

Approved for public release; distribution unlimited.

See additional restrictions described on inside pages

STINFO COPY

**AIR FORCE RESEARCH LABORATORY
MATERIALS AND MANUFACTURING DIRECTORATE
WRIGHT-PATTERSON AIR FORCE BASE, OH 45433-7750
AIR FORCE MATERIEL COMMAND
UNITED STATES AIR FORCE**

REPORT DOCUMENTATION PAGE				<i>Form Approved</i> OMB No. 0704-0188	
The public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Department of Defense, Washington Headquarters Services, Directorate for Information Operations and Reports (0704-0188), 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302. Respondents should be aware that notwithstanding any other provision of law, no person shall be subject to any penalty for failing to comply with a collection of information if it does not display a currently valid OMB control number. PLEASE DO NOT RETURN YOUR FORM TO THE ABOVE ADDRESS.					
1. REPORT DATE (DD-MM-YY) December 2006		2. REPORT TYPE Conference Proceedings Preprint		3. DATES COVERED (From - To)	
4. TITLE AND SUBTITLE DETECTION OF INCIPIENT THERMAL DAMAGE IN POLYMER MATRIX COMPOSITES (PREPRINT)				5a. CONTRACT NUMBER In-house	
				5b. GRANT NUMBER	
				5c. PROGRAM ELEMENT NUMBER 62102F	
6. AUTHOR(S) Eric Lindgren, John Welter, and Shamachary Sathish (AFRL/RXLP) Erik Ripberger (University of Dayton Research Institute)				5d. PROJECT NUMBER 4349	
				5e. TASK NUMBER RG	
				5f. WORK UNIT NUMBER M04R1000	
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Nondestructive Evaluation Branch (AFRL/RXLP) Metals, Ceramics & Nondestructive Evaluation Division Materials and Manufacturing Directorate Wright-Patterson Air Force Base, OH 45433-7750 Air Force Materiel Command, United States Air Force				8. PERFORMING ORGANIZATION REPORT NUMBER AFRL-RX-WP-TP-2008-4043	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) Air Force Research Laboratory Materials and Manufacturing Directorate Wright-Patterson Air Force Base, OH 45433-7750 Air Force Materiel Command United States Air Force				10. SPONSORING/MONITORING AGENCY ACRONYM(S) AFRL/RXLP	
				11. SPONSORING/MONITORING AGENCY REPORT NUMBER(S) AFRL-RX-WP-TP-2008-4043	
12. DISTRIBUTION/AVAILABILITY STATEMENT Approved for public release; distribution unlimited.					
13. SUPPLEMENTARY NOTES Conference paper submitted to the Proceedings of the 2007 Society for Advanced Materials and Processing Engineering (SAMPE) Conference. The U.S. Government is joint author of this work and has the right to use, modify, reproduce, release, perform, display, or disclose the work. PAO Case Number: AFRL/WS 07-0418, 25 Jan 2007.					
14. ABSTRACT Polymer matrix composite mechanical properties have been shown to decrease significantly with the presence of thermal damage. For aerospace applications, this type of damage typically occurs as a result of exposure to elevated temperatures from localized heating, such as lightning strikes, exhaust wash, or improper maintenance/repair procedures. Mechanical testing has shown that this type of damage, known as incipient damage, is present even when no visible damage is observable and can cause significant reduction in mechanical properties. Incipient damage is not currently readily detected with conventional nondestructive evaluation (NDE) tools. This presentation describes a NDE method that combines mechanical excitation with thermal imaging to detect the presence of surface and through-the-thickness incipient thermal damage without direct contact to the part being tested. It compares the results from samples with and without known damage using the thermo-elastic technique with similar inspection results from conventional NDE techniques, such as ultrasonic C-scan and thermography. These results indicate the thermo-elastic method identifies incipient damage that the other techniques fail to detect. In addition, an approach to analyze the thermo-elastic data to potentially determine the severity of the thermal damage is reviewed.					
15. SUBJECT TERMS					
16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT: SAR	18. NUMBER OF PAGES 14	19a. NAME OF RESPONSIBLE PERSON (Monitor) Dr. Kumar Jata 19b. TELEPHONE NUMBER (Include Area Code) N/A
a. REPORT Unclassified	b. ABSTRACT Unclassified	c. THIS PAGE Unclassified			

DETECTION OF INCIPIENT THERMAL DAMAGE IN POLYMER MATRIX COMPOSITES

Eric Lindgren, John Welter, Shamachary Sathish*, and Erik Ripberger**

Air Force Research Laboratory, Materials and Manufacturing Directorate, Nondestructive
Evaluation Branch, Wright-Patterson AFB, OH 45433

* *Air Force Research Laboratory, Materials and Manufacturing Directorate, Systems Support
Branch, Wright-Patterson AFB, OH 45433

*University of Dayton Research Institute, Dayton, OH 45469

ABSTRACT

Polymer matrix composite (PMC) mechanical properties have been shown to decrease significantly with the presence of thermal damage. This type of damage typically occurs as a result of exposure to elevated temperatures from localized heating, such as lightning strikes, exhaust wash, or improper maintenance/repair procedures. Studies have shown that a particular type of damage, known as incipient damage, is present even when no visible damage is observable and can cause significant reduction in mechanical properties. Incipient damage is not currently readily detected with conventional nondestructive evaluation (NDE) tools. This paper describes a NDE method that combines mechanical excitation with thermal imaging to detect the presence of surface and through-the-thickness incipient thermal damage without direct contact to the part being tested. It compares the results from samples with and without known damage using the thermo-elastic technique with similar inspection results from ultrasonic C-scan imaging. These results indicate the thermo-elastic method identifies volumetric incipient damage that the other techniques have extreme difficulty or fail to detect. In addition, an approach to analyze the thermo-elastic data to potentially determine the severity of the thermal damage is presented.

KEY WORDS: Nondestructive Evaluation, Degradation, Testing

1 INTRODUCTION / MOTIVATION

The use of non-metallic composite materials continues to increase, especially the use of PMC's in aerospace applications. The use of composites on the latest large commercial transports is approaching fifty percent by weight, whereas aluminum is less than fifteen percent by weight [1]. Typical advantages cited for using PMC's in aircraft structures include higher strength-to-weight ratios, plus enhanced corrosion and fatigue properties when compared to typical metallic

aerospace materials [1]. The damage modes found in PMC's are different when compared to metallic materials. For example, it is well known that impact damage can cause delaminations in the layers of PMC's. This type of damage can be readily detected by several inspection techniques, such as ultrasonics and thermography [2, 3]. These inspection methods typically use a single mode, such as a mechanical wave for ultrasound or a thermal gradient in thermography, to interrogate the material and receive some form of indication of damage.

Another source of potential damage to PMC's is thermal exposure. Thermal damage can be caused by accidental exposure to elevated temperatures beyond the use temperature of the matrix from a number of sources, such as lightning strikes, exhaust from engines or missiles, or through rare accidental misapplication of thermal blankets used in repair procedures [4]. Extreme exposure to excessive temperatures will yield damage that can be detected by conventional inspection techniques, such as ultrasonics and thermography. However, exposure to less intense thermal loads still causes damage in the PMC and may not be readily detected or exhibit any features that can be visibly detected. It has been shown in laboratory studies that PMC's can exhibit decreases in mechanical strength from ten to up to eighty percent from exposure to elevated temperatures, yet the damage is not visible and is not detected by traditional nondestructive evaluation (NDE) methods [5]. This type of damage is typically called incipient damage.

Several reviews have been performed to evaluate multiple NDE techniques to detect incipient thermal damage [6, 7]. According to these reviews, traditional NDE methods, such as ultrasonic, electromagnetic, and thermographic, can detect damage when it has progressed to delaminations or similar types of damage for both surface and sub-surface locations. However, these traditional techniques do not demonstrate promise for the detection of incipient damage. One method that has shown significant potential to detect incipient damage is called laser pumped fluorescence [8, 9]. This technique requires interaction between the laser beam and the damaged area, which requires that there is access to the surface to be inspected. Therefore, it is sensitive to only the surface damage and cannot provide information regarding subsurface damage.

Thus, the motivation for the efforts described in this paper is to explore an alternative NDE technique to detect volumetric incipient thermal damage in PMC's. The technique is based on using a high-powered ultrasonic signal to interrogate the potential damaged area and monitor this area with a thermal imaging camera to detect changes in the absorption of the mechanical ultrasonic energy. This method is based on similar approaches for detecting fatigue cracks in metallic structures. This method is typically called Sonic Infrared, or Sonic IR [10]. For metallic structures, the ultrasonic horn is in contact with the part being inspected. The thermal signature for the fatigue crack appears to be predominantly the result of friction at the crack surfaces, similar to the heat generated at the interface of a delamination when excited by vibrothermography [11]. Proof-of-principal of the thermo-elastic method explored in the reference indicates the ultrasonic horn does not need to be in contact with the sample when inspecting PMC's [12]. By using mechanical wave excitation, the changes in the behavior of the material due to incipient thermal damage can be detected. Therefore, this thermo-elastic technique combines two different NDE modalities, one for interrogation and another for detection.

This paper will expand on the proof-of-principal results to demonstrate the feasibility of measuring the changes of the thermo-elastic parameter in uniformly damaged PMC samples to detect the presence of incipient thermal damage. Two PMC samples were prepared and inspected before and after uniform thermal damage. A parametric study was performed to determine the effect of the changes in the excitation power on the inspection results and additional analysis was performed to determine if the thermo-elastic technique could provide quantifiable information regarding the severity of the incipient thermal damage. The results of the NDE inspections were compared to mechanical testing of damaged and undamaged samples.

2 TEST SAMPLES AND INDUCED DAMAGE

The samples used in this studied were prepared using a 16 ply lay-up consisting of Hexcel AS4 carbon fibers in a Cytec 977-3 epoxy resin. Cytec manufactured prepreg was used to prepare the samples and was cured according to the instructions provided by the supplier of this material. The 16 ply lay-up had the following quasi-isotropic configuration: $[0/+45/-45/90]_{2s}$. Two samples were prepared for the NDE experiments. These samples were prepared with a square geometry measuring 150 mm per side. Initial NDE inspections were performed on these two samples before exposing them to elevated temperatures. The samples were placed in a pre-heated oven and exposed to 274°C for 10 minutes and 20 minutes, respectively and cooled according to procedures preventing shock induced delaminations.

3 EXPERIMENTAL SET-UP AND MEASUREMENTS

Traditional ultrasonic inspections of the two samples before and after thermal exposure were performed in an ultrasonic immersion inspection tank. The samples were interrogated with a 5 MHz focused transducer with gates to detect the front-wall and back-wall reflectors, plus any additional reflections from the interior of the sample, which would be an indication of a delamination or other damage in the sample. The samples were raster scanned at approximately 0.5 mm increments to generate C-scan images of the inspection results.

A schematic of the instrumentation used for the thermo-elastic measurements is shown in Figure 1. The ultrasonic signal is transmitted into the sample using a 20 kHz ultrasonic horn. Note that the horn does not directly touch the sample, but is placed in close proximity to the sample surface. Thus, this signal is air-coupled to the sample, which eliminates any risk that the horn vibrations could generate damage in the composite sample. The ultrasonic signal locally heats the composite through thermo-elastic absorption. The temperature change in the sample is measured using a Merlin Mid infrared camera that has a sensitivity of 0.025°C. The camera is located on the opposite surface from the ultrasonic horn to measure the volumetric heating of the sample due to the thermo-elastic dampening. The temperature measurements for this study are relative and therefore the emissivity effects are cancelled. To perform out-of-plane displacement measurements, a fiber optic displacement sensor was used. The sensor replaced the infrared camera in the schematic shown in Figure 1. Environmental noise in the displacement sensor output was filtered using a high pass filter, which consisted of a low-noise pre-amplifier set with a gain of one. The amplitude of the displacement is measured using a digital oscilloscope.

Measurements were performed at six separate power levels of the ultrasonic horn before and after inducing thermal damage in the samples. At each power level, measurements were performed at five different locations on each sample and three measurements were performed at each location. The measurements before and after thermal exposure were performed at the same locations on the two samples. The fifteen data points from each power level were averaged to compare the changes in response as a function of the magnitude of the thermal damage in the two samples.

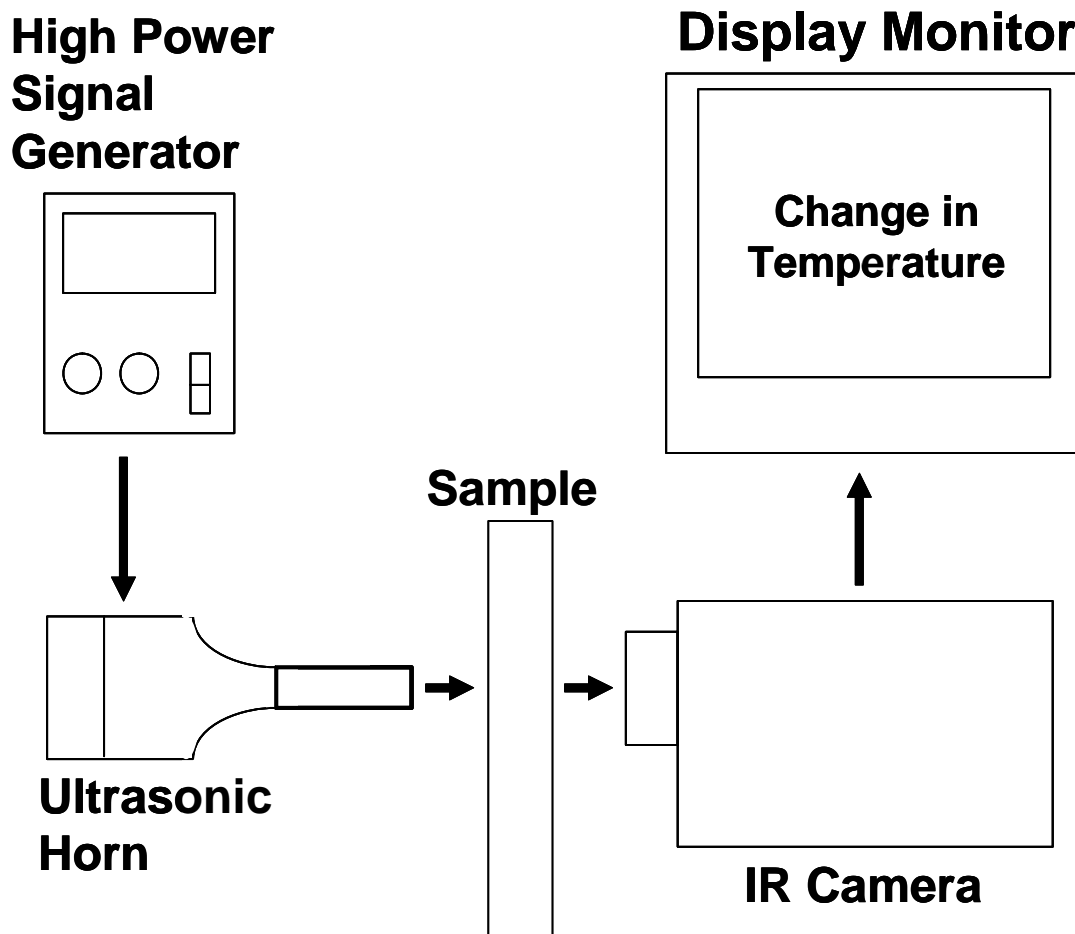


Figure 1. Schematic of experimental instrumentation used to measure the temperature changes in the samples as a function of excitation power before and after thermal damage.

Following the completion of the NDE measurements, both thermally exposed and undamaged samples were tested to determine the interlaminar shear strength according to ASTM Standard D-6272-02 [13]. The results of these mechanical tests were used to correlate the effect of the heat damage determined by the thermo-elastic measurements with the loss of mechanical strength.

4 RESULTS OF EXPERIMENTS – ANALYSIS AND DISCUSSION

The results from the ultrasonic C-scan inspections both before and after thermal exposure indicated there was no major damage in the samples, such as delaminations. Figure 2 shows the ultrasonic amplitude C-scan image for the sample exposed to 274°C for 20 minutes. There are several small reflectors in the sample that were correlated to surface features. No significant internal damage was detected in this sample, which represents the sample with the greatest amount of thermal exposure in this study.

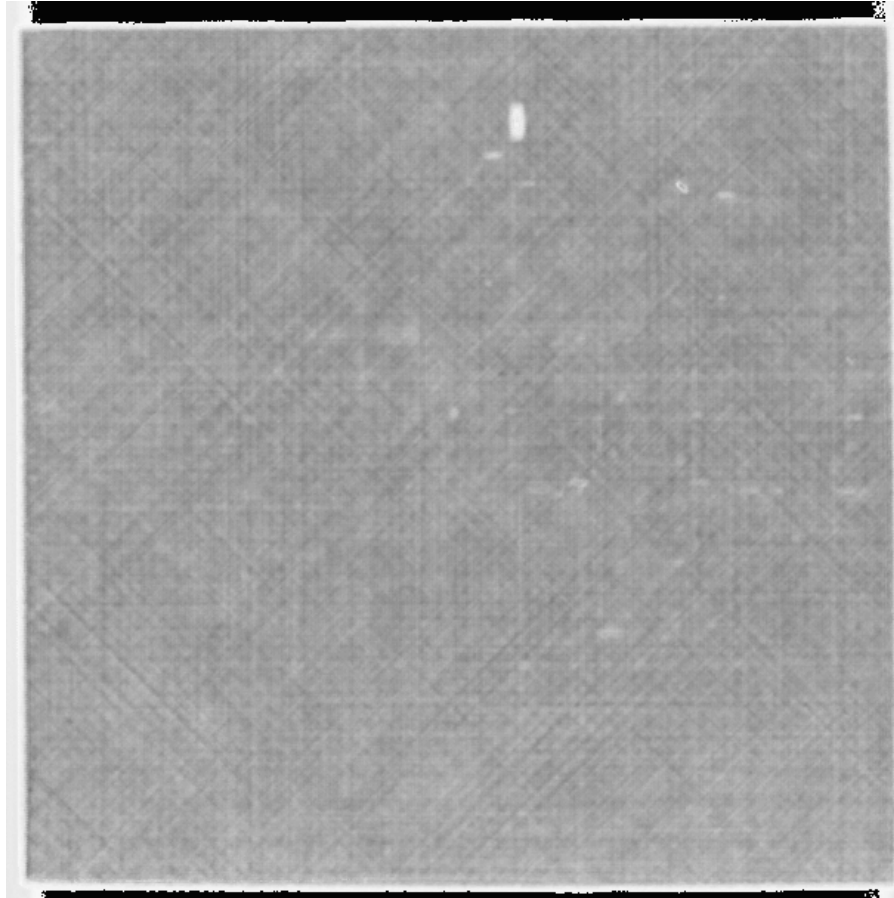


Figure 2. Amplitude ultrasonic C-scan image of the sample subjected to 20 minutes at 274° C.

The results from the interlaminar shear strength tests determined that the sample exposed to 10 minutes of the elevated temperature had an interlaminar shear strength reduction of 39 percent. For the sample exposed for 20 minutes, the decrease was 50 percent. These results, when combined with the ultrasonic C-scan results, confirm the thermal exposure was sufficient to induce incipient thermal damage in the composite samples, but was not sufficient to generate damage sufficiently severe to be detected visually or by ultrasonic C-scan inspections.

For the thermo-elastic measurements, the fifteen data points for each power level of the ultrasonic horn were averaged for the measured displacement of the sample surface and the temperature rise relative to the temperature before the application of the ultrasonic energy. The average temperature rise is plotted as a function of displacement for the six power levels. The results of plotting these data for the undamaged state and for the sample exposed to 20 minutes of elevated temperature are shown in Figure 3. Note that the temperature rise is less for the damaged sample when compared to the undamaged sample, whereas the magnitude of the displacement is greater for the damaged sample. This implies that the incipient thermal damage has generated a change in the matrix and/or matrix/fiber interface that allows more energy from the ultrasonic horn to be transmitted through the sample as less energy is being absorbed and converted to thermal energy by the damaged composite. Additional analysis is required to determine the degradation mechanism that correlates the decrease in interlaminar shear strength with the observed changes in the thermo-elastic behavior of the PMC.

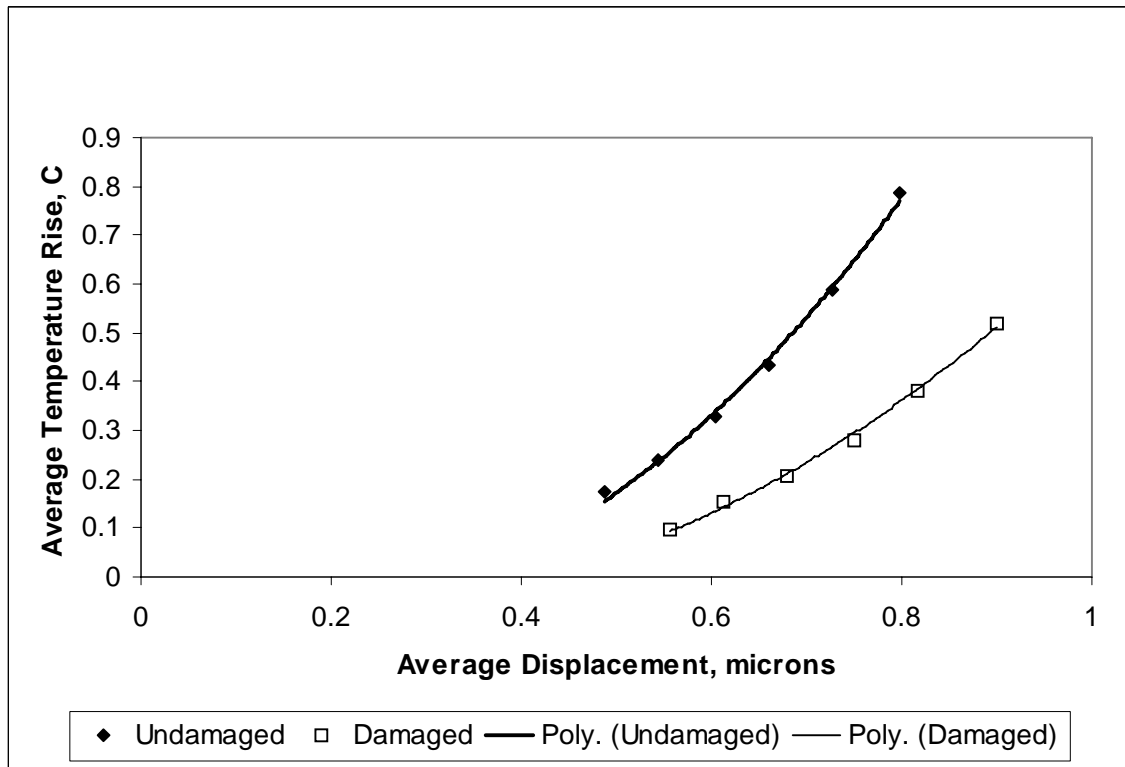


Figure 3. Plot of the average temperature rise as a function of sample displacement for six power levels of the ultrasonic horn for the sample exposed to 274°C for 20 minutes.

The curves shown in Figure 3, which are fitted to the experimental data, are quadratic. The change in the coefficients between the undamaged and damaged state for this graph is 43 percent. For the sample exposed to the same temperature for 10 minutes, the change in the coefficient was 29 percent. These results confirm the feasibility of using the change in the coefficients as a method to determine the severity of the incipient thermal damage present in PMC's. Additional

work on a broad range of samples would be required to determine the statistical scattering in these results, which will determine if this type measurement can be used to reliably quantify the amount of incipient thermal damage present in a polymer matrix composite.

5 SUMMARY

The results presented in this paper demonstrate the feasibility of using a thermo-elastic NDE technique to detect incipient volumetric thermal damage in PMC's, which has not been extensively demonstrated by other NDE techniques. The technique uses low frequency non-contact ultrasonic excitation to interrogate the PMC and measures the temperature changes generated by the thermo-elastic interaction in the PMC to determine if incipient thermal damage is present. The feasibility of this technique was demonstrated on two carbon fiber reinforced polymer matrix composites. The samples were 16 plies thick with a quasi-isotropic lay-up. Initial measurements, including ultrasonic C-scan images, were performed on the samples before they were thermally damaged by placing them in an oven at 274°C for 10 and 20 minutes. Ultrasonic C-scan images taken after the thermal exposure confirmed that no delaminations or other visible damage was present in the samples.

The samples were evaluated using the new thermo-elastic technique. Multiple locations of each sample were tested with six different power settings of the ultrasonic horn. The temperature changes in the samples as a result of this excitation were analyzed as a function of the displacement of the region of the composite excited by the ultrasonic horn. The quadratic relationship between the temperature change and sample displacement were found to change with increasing thermal exposure, indicating this technique was sensitive to the magnitude of incipient thermal damage in these samples. The presence of this damage was confirmed by measuring the interlaminar shear strength, which was found to decrease as a function of increasing thermal exposure. The analysis of the experimental data demonstrate the feasibility of using this technique to detect incipient thermal damage and indicates the potential to quantify the magnitude of the thermal damage before it can be detected by conventional NDE methods.

6 ACKNOWLEDGMENTS

The authors would like to acknowledge the assistance provided by Richard Riebel and Nicholas Kreitinger. This work was performed on-site in the NDE Branch, Materials and Manufacturing Directorate, Air Force Research Laboratory at Wright-Patterson AFB, OH with the support of the on-site contract number F33615-03-C-5219.

7 REFERENCES

1. R. Griffiths, "Boeing sets pace for composite usage in large civil aircraft," <http://www.compositesworld.com/hpc/issues/2005/May/865/1>
2. A.C. Wey and L.W. Kessler, "Ultrasonic Imaging of Damage Progression in Composite Laminates," *Proceedings of the 1992 Ultrasonics Symposium*, p777, (1992).
3. E.A. Lindgren, et. al.. *Proceedings of SPIE Thermosense*, (1997).

4. C.J. Janke, et al, "Composite Heat Damage Assessment," Conference on Characterization and NDE of Heat Damage in Graphite Epoxy Composites, NTIAC, pp 76-96, 1993
5. Frame, B. J., et al, "Composite Heat Damage, Part 1. Mechanical Testing of IM6/3501-06 Laminates, Part 2. Nondestructive Evaluation Studies of IM6/3501-06 Laminates," ORNL/ATD-33, Oak Ridge National Laboratory, Oak Ridge, TN, 1990
6. G. A. Matzkanin, "Nondestructive Characterization of Heat Damage in Graphite/Epoxy Composite: A State-of-the-Art Report," <http://www.ntiac.com/gamsoar.html>
7. G. A. Matzkanin, and G. P. Hansen "Heat Damage in Graphite Epoxy Composites: Degradation, Measurement and Detection: A State-of-the-Art Report," NTIAC, (1998).
8. W. G. Fisher, K. E. Meyer, E. A. Wachter, D. R. Perl and P. J. Kulowitch, Mater. Eval., Sept 1997, p726
9. G. L. Powel, N. R. Smyrl, C. J. Janke, E. A. Watcher, W. G. Fisher, J. Lucania, M. Milosevic and G. Auth, "Nondestructive Inspection of Graphite-Epoxy Laminates for Heat Damage Using Drift and LPF Spectroscopies," Conference on Conference on Characterization and NDE of Heat Damage in Graphite Epoxy Composites, NTIAC, 1993.
10. X. Han, W. Li, Z. Zeng, L.D. Favro, and R.L. Thomas, "Acoustic Chaos and Sonic Infrared Imaging," Applied Physics Letters, **81**, 17, 3188, (Oct, 2002)
11. E.G. Henneke, K.L. Reifsnider, W.W. Stinchcomb, "Thermography, an NDI Method for Damage Detection," Journal of Metals, pp 11-15, (Sept. 1979).
12. S. Sathish, J. Welter, R. Reibel, C. Buynak, "Thermo-elastic Characterization of Heat Damage in Carbon Fiber Epoxy Composites," Rev. Prog. QNDE, **25**, p1015, (2005).
13. C.E. Browning, F.L. Abrams, and J.M. Whitney, "A Four-Point Shear Test for Graphite/Epoxy Composites," Special Technical Publication 787, p54, ASTM, (1983).